

WHAT IS CLAIMED IS:

1. Apparatus for etching a sample, said apparatus comprising:  
(a) a source of etchant gas;  
(b) an etching chamber in communication with said source of etchant gas;  
(c) a recirculation loop passing through said etching chamber; and  
(d) a pump disposed within said recirculation loop for recirculating etchant gas along said recirculation loop.

2. Apparatus in accordance with claim 1 in which said source of etchant gas comprises a source chamber.

3. Apparatus in accordance with claim 2 further comprising an expansion chamber communicating with said source chamber and with a gas source for a gas other than said etchant gas, said expansion chamber arranged for mixing gas from said source chamber with gas from said gas source.

4. Apparatus in accordance with claim 3 in which said expansion chamber is in communication with said recirculation loop.

5. Apparatus in accordance with claim 1 further comprising a filter disposed within said recirculation loop, said filter being one that removes a member selected from the group consisting of byproducts or effluent from gases flowing through said recirculation loop, or particulates.

6. Apparatus in accordance with claim 1 in which said pump is a dry pump.

7. Apparatus in accordance with claim 6 in which said dry pump has no wet seals and adds no gas to said recirculation loop.

5 8. Apparatus in accordance with claim 7 in which said dry pump is a bellows pump.

9. Apparatus in accordance with claim 8 in which said bellows pump comprises a housing with bellows-type wall sections enclosing a hollow interior, and at least one partition disposed to divide said hollow interior into a plurality of sections.

10 10. Apparatus in accordance with claim 1 in which said pump is constructed to circulate etchant gas substantially continuously within said recirculation loop.

15 11. Apparatus in accordance with claim 3 in which said pump is defined as a first pump and said apparatus further comprises a second pump arranged to draw gases from a member selected from the group consisting of said expansion chamber, said source chamber, and said recirculation loop.

20 12. Apparatus in accordance with claim 2 further comprising gas flow spreading means in said source chamber for diverting incoming gas.

13. Apparatus in accordance with claim 12 in which said gas flow spreading means is a baffle.

25 14. Apparatus in accordance with claim 12 in which said gas flow spreading means is a perforated plate.

15. Apparatus in accordance with claim 1, further comprising an energy source and/or electric field source at the etching chamber for forming a plasma therein.

5 16. Apparatus in accordance with claim 2 in which said source of etchant gas further comprises fluoride crystals retained within said source chamber.

17. Apparatus in accordance with claim 16 in which said fluoride crystals are xenon difluoride crystals.

10 18. Apparatus in accordance with claim 3 in which said gas source for a gas other than said etchant gas comprises a source of a gas with molar averaged molecular weight less than or equal to that of N<sub>2</sub>.

15 19. Apparatus in accordance with claim 18 in which said gas other than said etchant gas is a member selected from the group consisting of Ar, Ne, He and N<sub>2</sub>.

20 20. Apparatus in accordance with claim 3 in which said gas source for a gas other than said etchant gas comprises a plurality of gas sources, the gases from which, when mixed, yield a gaseous mixture with molar averaged molecular weight less than or equal to that of N<sub>2</sub>.

21. Apparatus in accordance with claim 20 in which said plurality of gas sources are sources of two or more members selected from the group consisting of Ar, Ne, He and N<sub>2</sub>.

25 22. A method for etching a sample, said method comprising:  
(a) placing said sample in an etching chamber disposed within a gas recirculation loop, said etching chamber

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in communication with a source of etchant gas, and  
said gas recirculation loop having a pump disposed  
therein;

- (b) passing etchant gas from said source of etchant gas into said etching chamber to expose said sample to said etchant gas; and
- (c) recirculating said etchant gas through said recirculation loop by way of said pump.

10 23. A method in accordance with claim 22 further comprising passing said etchant gas through an expansion chamber prior to step (b) and, while said etchant gas is in said expansion chamber, forming a mixture of said etchant gas with non-etchant gases, and step (b) comprises passing said etchant gas as part of said mixture into said etching chamber.

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20 24. A method in accordance with claim 22 in which said pump is a continuous recirculation pump and step (c) comprises continuously recirculating said etchant gas through said recirculation loop.

25. A method in accordance with claim 22 further comprising bleeding etchant gas into said recirculation loop during step (c).

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26. A method comprising:  
providing an apparatus according to claim 1;  
providing a solid or liquid etchant at said etchant source at a temperature and pressure sufficient to cause said etchant to vaporize;

providing a sample to be etched within the etching chamber;

passing the vaporized etchant through the etching chamber; and

recirculating the etchant multiple times through the etching chamber with said pump.

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27. A method in accordance with claim 26, wherein the etchant gas is passed through the pump without additional gas being added thereto.

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28. A method in accordance with claim 26, wherein the source of etchant gas comprises two chambers, wherein the temperature and/or pressure of one chamber is different from the pressure and/or temperature of the other so that predominantly liquid or solid etchant remains in one chamber and predominantly gas etchant is in the other, prior to passing into the recirculation path and etching chamber.

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29. A method in accordance with claim 26 comprising heating the process gas so as to at least avoid condensation, and cooling the etching chamber and/or sample to improve selectivity between the silicon and non-silicon portions of the sample.

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30. A method in accordance with claim 26 in which said sample comprises a silicon portion existing in at least one layer and one or more non-silicon portion existing in at least one layer, said silicon etchant is a fluoride gas selected from the group consisting of noble gas fluorides and halogen fluorides, and said gas is a gas mixture which further comprises a non-etchant gas additive at a partial pressure and a molar ratio relative to said fluoride gas such that said gas mixture achieves substantially greater etching selectivity

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toward said silicon portion than would be achieved with said fluoride gas alone.

5        31. A method in accordance with claim 30 in which said non-etchant gas additive is a member selected from the group consisting of nitrogen, argon, helium, neon, and mixtures thereof.

10      32. A method in accordance with claim 30 in which said non-etchant gas additive is a member selected from the group consisting of helium, a mixture of helium and nitrogen, and a mixture of helium and argon.

15      33. A method in accordance with claim 30 in which said fluoride is a xenon fluoride and said non-etchant gas additive is helium.

20      34. A method in accordance with claim 30 in which said non-silicon portion is a member selected from the group consisting of titanium, gold, tungsten, and compounds thereof.

25      35. A method in accordance with claim 30 in which said silicon portion is a silicon layer deposited over a substrate and said non-silicon portion is a layer of a member selected from the group consisting of silicon nitride, silicon carbide, and silicon oxide, deposited over said silicon layer, said non-silicon layer being patterned to leave vias therein for access of said gas to said silicon layer, the exposure of said sample to said gas being of sufficient duration to laterally etch away substantially all of said silicon layer by access through said vias.

36. A method in accordance with claim 26 in which said sample is a substrate for a member selected from the group consisting of a semiconductor and/or a MEMS device.

5 37. A method in accordance with claim 26 in which said sample is a substrate for a MEMS device.

10 38. Apparatus for exposing a silicon-containing sample to a gas comprising a gaseous fluoride etchant for etching silicon, said apparatus comprising:  
a flow-through etching chamber comprising:  
a sample support,  
entry and exit ports for said gas;

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a source chamber comprising a fluoride etchant in solid or liquid form, the source chamber and the etching chamber capable of being in fluid communication with each other;  
a recirculation loop and recirculation pump within the loop, the recirculation loop constructed to connect to the etching chamber at two locations to allow etching gas to flow into and out of the etching chamber, and the recirculation pump in communication with the etching chamber and adapted to pump etching gas repeatedly through the etching chamber.

20 39. Apparatus in accordance with claim 38 further comprising a baffle and perforated plates comprising parallel circular plates arranged coaxially within said flow-through chamber.

25 40. Apparatus in accordance with claim 39 in which said perforations in said perforated plate are of decreasing diameter from the center of said perforated plate outward.

30 41. Apparatus in accordance with claim 40, further comprising a plasma generator at said etching chamber.

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42. A reciprocating pump comprising:  
an enclosed housing comprising bellows-type wall sections  
and a partition arranged to divide the interior of said  
housing into first and second chambers, said partition  
being movable in a reciprocating manner to cause  
collapse and extension of said bellows-type wall  
sections whereby one chamber contracts while the  
other expands and vice versa;  
inlet and outlet ports for each chamber with controllable  
shutoff valves at each port; and  
a partition driver for moving said partition in a reciprocating  
manner and opening and closing said shutoff valves  
in a coordinating sequence, causing said chambers  
to draw fluid in through alternating inlet ports while  
discharging fluid through alternating outlet ports and  
thus together to produce a substantially continuous  
outlet flow.

43. A reciprocating pump in accordance with claim 42 in  
which all surfaces of said bellows-type wall sections, said  
partition, and any other components that face the interior of  
said chambers are of material that is resistant to corrosion  
by noble gas fluorides and halogen fluorides.

44. A reciprocating pump in accordance with claim 42 in  
which said chambers are sized and said partition driver is  
selected to achieve a pumping rate of from about 3 liters per  
minute to about 300 liters per minute.

45. Apparatus for etching silicon from a sample by  
exposing said sample to a gas comprising a silicon etchant,  
said apparatus comprising:

a flow-through chamber having:  
a sample support,

entry and exit ports for said gas,  
a perforated plate between said entry port and said  
sample support, and  
a baffle between said entry port and said perforated plate,  
5 said baffle positioned to deflect said etchant gas from  
said etchant port radially toward the periphery of said  
perforated plate, and said perforated plate containing  
an array of perforations arranged to distribute said  
deflected etchant gas over all exposed surfaces of  
10 said sample; and  
a reciprocating pump driving said gas toward said entry port,  
said  
reciprocating pump comprising:  
15 an enclosed housing comprising bellows-type wall sections  
and a partition arranged to divide the interior of said  
housing into first and second chambers, said partition  
being movable in a reciprocating manner to cause  
collapse and extension of said bellows-type wall  
sections whereby one chamber contracts while the  
other expands and vice versa;  
20 inlet and outlet ports for each chamber with controllable  
shutoff valves at each port; and  
a partition driver for moving said partition in a reciprocating  
manner and opening and closing said shutoff valves  
25 in a coordinating sequence, causing said chambers  
to draw fluid in through alternating inlet ports while  
discharging fluid through alternating outlet ports and  
thus together to produce a continuous outlet flow.

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46. Apparatus in accordance with claim 45 in which said  
30 reciprocating pump draws gas from said exit port.

47. Apparatus for adding or removing a layer of material from a sample by contacting said sample with a process gas, said layer having at least one dimension less than 1mm, said apparatus comprising:

5 (a) a source of said process gas;

(b) a fabrication chamber in communication with said source of process gas;

(c) a recirculation loop passing through said fabrication chamber; and

10 (d) a pump disposed within said recirculation loop for recirculating process gas along said recirculation loop.

48. Apparatus in accordance with claim 47 in which said process gas corrodes metal in the presence of moisture.

15 49. Apparatus in accordance with claim 48 in which said moisture is water moisture.

50. Apparatus in accordance with claim 47 further comprising a filter disposed within said recirculation loop, said filter being one that removes a member selected from the group consisting of byproducts, particulates or effluents from gases flowing through said recirculation loop.

20 51. Apparatus in accordance with claim 47 in which said source of process gas is comprised of a member selected from the group consisting of (i) chamber retaining a said process gas and a condensed liquid phase of said process gas in equilibrium with said process gas, (ii) a pressurized chamber of said process gas, and (iii) a chamber retaining a solid condensed phase of said process gas.

25 52. Apparatus in accordance with claim 47 in which said source of process gas is comprised of first and second

chambers, said first chamber retaining primarily a liquid or solid condensed form of said process gas, and said second chamber retaining said process gas evaporated or sublimed from said condensed form, said first and second chambers being maintained at different temperatures.

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53. Apparatus in accordance with claim 51 further comprising a source of pressurized diluent gas and an expansion chamber positioned to receive diluent gas from said source of diluent gas and process gas from said source of process gas and to mix said diluent gas and said process gas thus received.

54. Apparatus in accordance with claim 47 in which said layer has at least one dimension less than 500 $\mu\text{m}$ .

55. Apparatus in accordance with claim 47 in which said layer has at least one dimension less than 100 $\mu\text{m}$ .

56. Apparatus for etching a sample by contacting the sample with a vapor fluoride etchant gas:

(a) a source of said fluoride etchant gas, said source of etchant gas being comprised of first and second chambers, said first chamber retaining primarily a liquid or solid condensed form of said fluoride etchant gas, and said second chamber retaining said fluoride etchant gas volatilized from said condensed form, said source comprising a temperature regulator for maintaining the first and second chambers at different temperatures;

(b) an etching chamber in communication with said source of fluoride etchant gas for holding the sample to be etched by the fluoride etchant gas.

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~~57~~ 58. Apparatus in accordance with claim ~~57~~, in the absence of a source of energy for energizing the etchant gas once in gas form.

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~~58~~ 59. Apparatus in accordance with claim ~~57~~, wherein the first source chamber is held at a temperature less than the second source chamber.

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~~59~~ 60. Apparatus in accordance with claim ~~59~~, wherein the two source chambers are maintained at more than 3 degrees C difference.

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~~60~~ 61. Apparatus in accordance with claim ~~59~~, wherein both source chambers are maintained at temperatures under 40 degrees C.

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~~61~~ 62. Apparatus in accordance with claim ~~58~~, further comprising a recirculation path for recirculating the fluoride etchant gas repeatedly through the etching chamber.

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~~62~~ 63. Apparatus in accordance with claim ~~57~~, wherein the first source chamber comprises primarily liquid or crystals of a halogen or noble gas fluoride.

~~63~~ 64. Apparatus in accordance with claim ~~57~~, further comprising a cooling unit for cooling the process gas, one or more of the aforementioned chambers and/or the sample being etched.

~~64~~ 65. Apparatus in accordance with claim ~~64~~, wherein the cooling unit is adapted to cool the process gas, one or

more of the aforementioned chambers and/or sample below room temperature.

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~~65~~ 66. Apparatus in accordance with claim ~~65~~, wherein the cooling unit is adapted to cool in the range of from about 1 to 15 degrees C.

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~~66~~ 67. Apparatus in accordance with claim ~~57~~, wherein the sample comprises silicon and one or both of a dielectric and a metal, and the silicon is etched relative to the dielectric and/or metal.

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~~67~~ 68. Apparatus in accordance with claim ~~67~~, wherein the dielectric is a silicon nitride or silicon oxide layer.

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68. Apparatus for etching a sample comprising a silicon material and a dielectric material, comprising:  
a source of a noble gas halide and/or halogen halide etchant gas;  
an etching chamber in communication with the source of the etchant gas;  
a surface within the etching chamber for holding the sample to be etched;  
a cooling unit for cooling the surface, etching chamber and/or etchant gas below room temperature.

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69. Apparatus according to claim ~~68~~, wherein the source comprises a source chamber having therein a liquid or solid noble gas halide or halogen halide.

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70 71. Apparatus according to claim 70, wherein the source chamber comprises xenon difluoride crystals and/or bromine trifluoride liquid.

71 72. Apparatus according to claim 71, comprising a second source chamber connected to said source chamber and maintained at a temperature higher than the temperature of said source chamber.

72 73. Apparatus according to claim 69, further comprising a sample held by a holder, the sample comprising a sacrificial silicon portion and a dielectric portion.

73 74. A method in accordance with claim 26, wherein the etchant gas is passed through a baffle and a perforated plate within the etching chamber.

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